
Paints and varnishes — Wettability —
Part 6:
Measurement of dynamic contact angle

Peintures et vernis — Mouillabilité —

Partie 6: Mesurage de l'angle de contact dynamique

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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

A list of all parts in the ISO 19403 series can be found on the ISO website.

Introduction

Dynamic contact angles describe the processes on the interface liquid/solid during volume increase (advancing angle) or volume decrease (receding angle) of a drop in horizontal position. As an alternative to the static method (see ISO 19403-2), for the advancing angle always a surface area is wetted, which was previously unwetted. For the receding angle, the contact angle during dewetting is observed. The difference between advancing angle and receding angle is a sign of different chemical or physical homogeneity (morphology, topology) or roughness. The receding angle is not suitable for the determination of the surface energy.

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Paints and varnishes — Wettability —

Part 6: Measurement of dynamic contact angle

1 Scope

This document specifies a method to measure the dynamic contact angle with an optical method. The advancing and the receding angles are determined.

By means of this defined measurement, the wetting and dewetting properties can be characterized. It can also be concluded on the morphological and chemical homogeneity of interfaces.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 4618, *Paints and varnishes — Terms and definitions*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

ISO 19403-1, *Paints and varnishes — Wettability — Part 1: Terminology and general principles*

ISO 19403-2:2017, *Paints and varnishes — Wettability — Part 2: Determination of the free surface energy of solid surfaces by measuring the contact angle*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4618 and ISO 19403-1, and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

3.1 dynamic contact angle

contact angle, which is measured during advancing or receding of the three-phase point

Note 1 to entry: For the definition of “contact angle”, see ISO 19403-1:2017, 3.1.9.

Note 2 to entry: The advancing or receding of the three-phase point can be achieved by changing the volume of the liquid drop to be measured, by relative movement (immersing and pulling out) of a solid body to an interface, or by moving the drop over the interface (e.g. rolling off).

3.2 advancing angle

θ_a

contact angle, which is measured during advancing of the three-phase point

Note 1 to entry: Generally, the advancing angle is used for the determination of the interface energy, in which case, the measurement should be carried out close to the thermodynamic equilibrium. This is approximately reached if there is no influence of, for example, the dosing speed on the contact angle.

3.3 receding angle

θ_r

contact angle, which is measured during receding of the three-phase point

3.4 contact angle hysteresis

θ_{ar}

difference between *advancing angle* (3.2) and *receding angle* (3.3)

3.5 polynomial method

<contact angle> image-analysing evaluation method for the contact angle which can also be applied when the dosing needle is still inside the drop

4 Principle

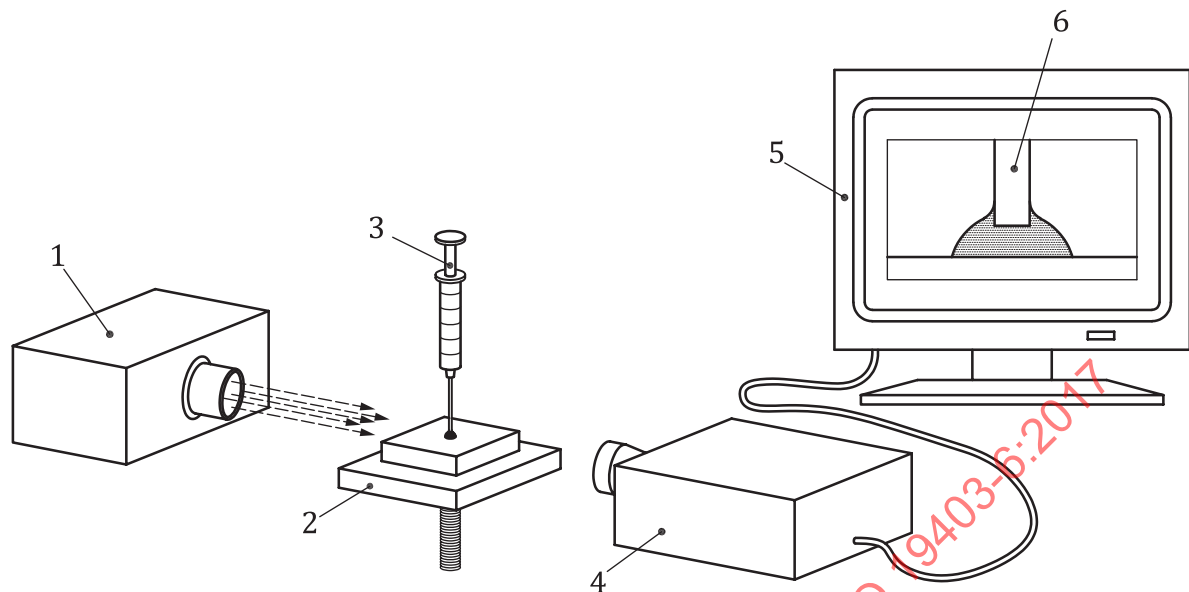
At least three drops of the respective test liquids are dosed onto the flat surface of a test specimen. The volume of the respective drop is continuously increased (advancing angle) or decreased (receding angle). The contact angle is preferably determined by means of the polynomial method, synchronously with the dosing. If the polar and dispersive fraction of the surface free energy is to be determined according to ISO 19403-2, the advancing angle shall be used.

5 Apparatus and materials

Ordinary laboratory apparatus, together with the following.

5.1 Contact angle measuring system.

Any state-of-the-art contact angle measuring device, preferably systems with digital image capture and analysis for measuring the contact angle. [Figure 1](#) shows a schematic example of a contact angle measuring system.



Key

- 1 light source
- 2 specimen holder
- 3 system with microlitre syringe for continuous dosing
- 4 optical system
- 5 screen
- 6 needle positioned in the drop

Figure 1 — Schematic diagram of a contact angle measuring system

The image capturing system should be oriented in a way that the optimal image resolution ratio (ratio of width and height) can be used.

NOTE The device used can differ from the schematic diagram in regard to light path and the arrangement of the components.

5.2 Dosing unit.

Dosing unit, which makes it possible to continuously change the drop volume on the surface in the range of microlitres.

NOTE Typical dosing rates for test liquids for the determination of the surface energy are in the range of 10 µl/min.

5.3 Test liquids.

The test liquids shall not physically or chemically affect the surface. They shall not have a distinct yield point.

NOTE A notable yield value is shown when a lamella of the liquid teared with a needle does not level within a given time limit (e.g. 30 s).

The test liquids shall not crosslink during measuring, not form skins and not volatilize distinctly.

Liquids having a vapour pressure higher than water at 30 °C shall be measured in the saturated vapour phase.

6 Sampling

Take a representative specimen of the solid substrate to be tested as described in ISO 15528. The specimens shall not be contaminated before measuring.

For advice on sampling and sampling preparation, see also [Annex A](#).

7 Procedure

7.1 General for measuring on the horizontal drop

7.1.1 Setting up the contact angle measuring system

Choose the location of the contact angle measuring system so that it is not exposed to

- vibrations,
- intense air flows (e.g. caused by air conditioning), and
- intense exposure to light from outside (e.g. windows, bright lighting).

Align the contact angle measuring system horizontally.

7.1.2 Test conditions

Carry out the test at $(23 \pm 2) ^\circ\text{C}$ and a relative humidity of $(50 \pm 5) \%$ (see ISO 3270) and make sure that all test media have this temperature.

7.1.3 Conditioning of the test panels

Condition the test panels at a temperature of $(23 \pm 2) ^\circ\text{C}$ and a relative humidity of $(50 \pm 5) \%$ for a minimum of 16 h prior to testing. Carry out the test immediately after conditioning.

7.1.4 Conditioning of the test liquids

Condition the liquids in a closed container at a temperature of $(23 \pm 2) ^\circ\text{C}$ prior to testing. Carry out the test immediately after conditioning.

7.2 Measurement

7.2.1 General

Place a preferably flat test specimen of the surface to be measured on the specimen holder. Adjust the specimen holder so that the surface of the test specimen is located in the lower half of the image and that it is horizontally aligned.

Fill the dosing system with the chosen liquid. Pay attention to fill without contamination or bubbles.

Adjust an image representation that is sufficient in regard to brightness and contrast (mind the specifications given by the manufacturer).

If possible, adjust the light source of the contact angle measuring device so that the grey values within the drop close to the phase interface do not exceed the value 40 (referring to 256 grey value grades) and amount to a minimum of 170 on the outside of the drop.

NOTE It can be reasonable to test the modes of operation of the optical components by means of two-dimensional images of drops. Such reference images are commercially available.

Bring the needle into focus. Adjust the zoom of the contact angle measuring device so that the maximum width of the contour of the drop is imaged entirely at maximum expansion.

7.2.2 Measuring method

Choose the distance between the dosing needle and the surface so that the influence on the expected contour of the drop is as slight as possible.

NOTE 1 As a first guidance for the distance between the needle and the surface of the test specimen, the one-and-a-half-times diameter of the needle can be used.

Especially for low contact angles, minimize the pull-up of liquid on the needle, if necessary, by using a needle of poorly wettable material.

Choose the dosing speed as slow as possible so that the contact angle of the drop is as close to the thermodynamic equilibrium contact angle as possible.

Typical dosing speeds for the determination of the surface energy are in the range of 10 µl/min. For test liquids with higher viscosity than most of the test liquids given in ISO 19403-2:2017, Table 1, the dosing speeds shall be reduced. This especially applies for glycerol.

NOTE 2 It is common to start measuring only after a dosing of 3 µl minimal volume.

NOTE 3 Due to the limited image section and the limited precision, it can be difficult to measure advancing angles below 10° by means of the dynamic method.

Align the baseline so that it runs through the three-phase points of the drop.

NOTE 4 A top-view angle of the camera to the horizontal can be adjusted to help find the three-phase points. The top-view angle causes an image error of the drop projection. This may have an influence on the measuring result of the contact angle and can be corrected.

Start measuring the contact angle immediately after dosing the minimal volume. Record the measuring values as function of time.

NOTE 5 In comparison to the measuring method of the static contact angle, interfering transformations of material often occur less in regard to the dynamic contact angle. This especially applies for short measuring times.

7.2.3 Determination of the contact angle

Preferably, determine the advancing and receding angle by means of the polynomial method.

Measure on a minimum of three different measuring points on the test specimen in order to obtain sufficient information in regard to the homogeneity of a test specimen. Previously wetted positions shall not be used. Arguable readings which may be caused by dust, contaminations, etc. shall not be included in the calculation of the mean value. Discarded, possibly contaminated test liquid shall not be reused.

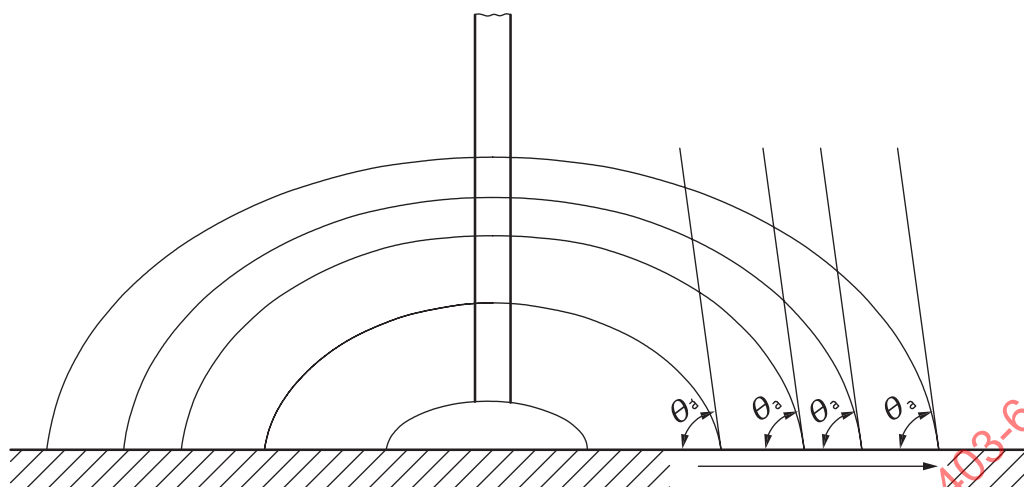
Repeat the measuring with at least one more liquid, which was selected in accordance with the criteria given in 5.3.

8 Evaluation

Present the measured contact angles as function of time. Determine the advancing and receding angle from these data (see [Figure 2](#) and [Figure 3](#)). An advancing and receding angle can only be described if a respective plateau forms in the contact angle/time diagram (see [Figure 4](#)). This requires that the measured advancing or receding angle remains constant over a certain time period or at least varies around a mean value.

The wettability behaviour is characterized by the advancing angle. The dewettability behaviour is characterized by the receding angle.

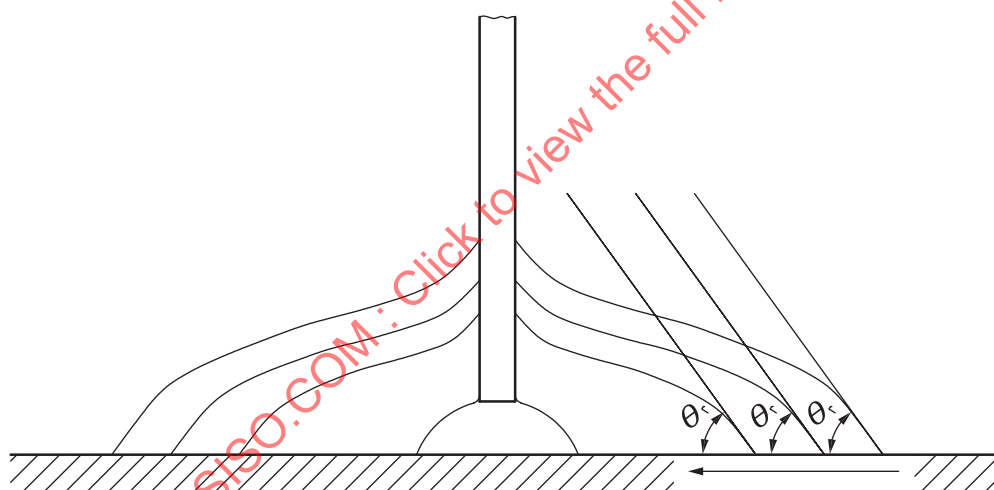
The method for determining the surface free energy is specified in ISO 19403-2.



Key

θ_a advancing angle

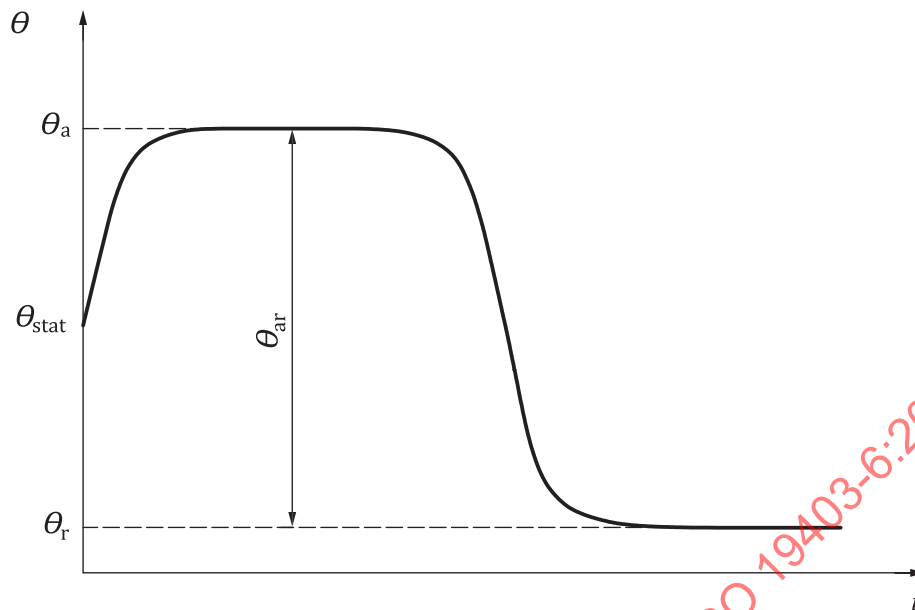
Figure 2 — Applying a drop through a needle for the dynamic measurement of the advancing angle



Key

θ_r receding angle

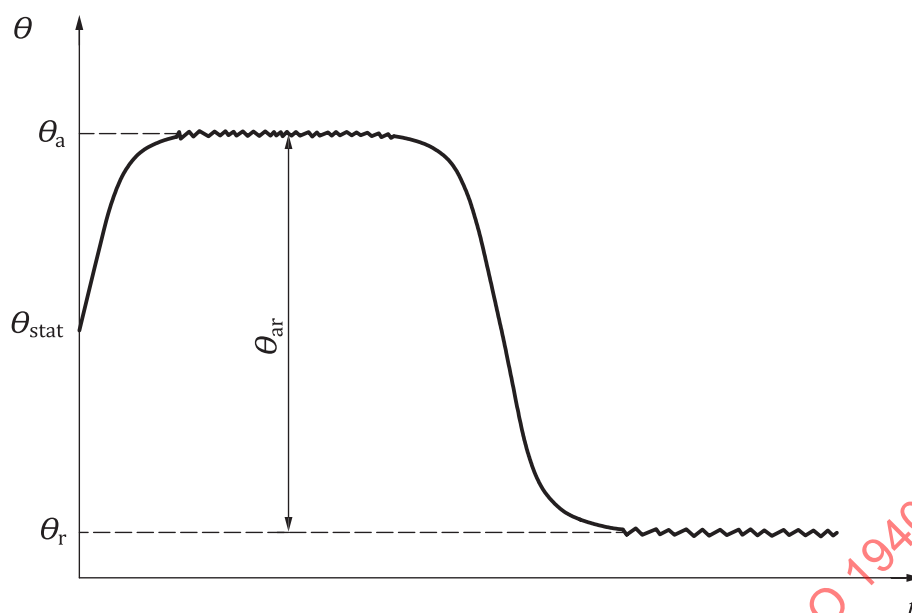
Figure 3 — Extracting a drop through a needle for the dynamic measurement of the receding angle

**Key**

- θ dynamic contact angle
- θ_a advancing angle
- θ_{stat} static contact angle
- θ_r receding angle
- θ_{ar} contact angle hysteresis
- t time

Figure 4 — Time curve of the contact angle during advancing and receding measuring

In the case of rough and chemically inhomogeneous surfaces, severe time variations of the advancing angle can occur. These occur when the diameter of the drop does not change even though the volume of the drop is continuously increased. This results in an increase of the contact angle until this contact angle is decreased instantly by the abrupt expansion of the drop. This phenomenon is called slipstick (see [Figure 5](#)).



Key

- θ dynamic contact angle
- θ_a advancing angle
- θ_{stat} static contact angle
- θ_r receding angle
- θ_{ar} contact angle hysteresis
- t time

Figure 5 — Slipstick behaviour of the time curve of the contact angle during advancing and receding measuring

9 Precision

At the time of publication, information on precision is not available.

10 Test report

The test report shall contain at least the following information:

- a) all details necessary to identify the test specimen (manufacturer, product identification, batch number, etc.);
- b) a reference to this document, i.e. ISO 19403-6;
- c) the used test liquids;
- d) the used drop volumes at the beginning and upon completion of measurement;
- e) the dosing speed and waiting times, if necessary;
- f) the method with which the contact angle of the drop was obtained, if deviating from the polynomial method;
- g) the top-view angle and, if necessary, the used correction;
- h) the contact angle/time diagram;